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RESEARCH ARTICLE - SPECIAL ISSUE - IMPACT OF ADVANCED TRIBOLOGICAL TECHNOLOGIES ON MODERN INDUSTRY

Comparative Mechanical, Tribological and Morphological Properties of Epoxy Resin Composites Reinforced With Multi-Walled Carbon Nanotubes

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Abstract

Addition of multi-walled carbon nanotubes (MWCNTs) guarantees enhancement of the characteristics offered in the polymer composite materials with negligible weight increase. In this research work, ultrasonication and compression moulding technique were used for the manufacturing of MWCNT–epoxy composites. Five formulations of epoxy composites of 5.0 ± 0.1 mm thickness with symmetrical geometry were manufactured. This research focuses on analysing the mechanical (tensile, flexural, impact and hardness measurements), tribological (with the help of Pin-on-Disc tribometer) and morphological [by using scanning electron microscopy and transmission electron microscopy (TEM)] properties on MWCNT–epoxy composites. The MWCNTs were blended in resins having 0 (neat sample), 0.5, 1, 1.5 and 2 wt%. The results showed that 0.5 wt% of MWCNT–epoxy composite possesses improved properties, while further increment in concentration of MWCNT had detrimental effect on the properties. It was also noted from the TEM analyses that intense interactivity exists among the epoxy and MWCNTs and this further assists in improving the interfacial bonding strength among the resin and nanoparticles. The results of this work were adequate in the fields where greater mechanical, tribological and morphological properties were expected, like in structural, aerospace and automobiles industries.

Keywords MWCNT · Epoxy composites · Mechanical · Tribology · Morphology

1 Introduction

Recently, importance of the synthesis and properties of nanoparticles had progressively grown because of the great potential for their applications in different fields of material science and technology [1, 2]. This was due to their exclusive superior properties such as mechanical, optical, catalytic, magnetic, anti-friction and anti-wear [3]. The evolution of carbon nanotubes (CNTs) had led to their application in the development of different composite materials with varying properties [4]. Kaya and Parlar [5] investigated that the variations in the reinforcement material, its direction and the matrix material type had an impact on different tribological behaviour of composites. CNTs had been applied as a mechanical property enhancer and wear reduction reinforcement for epoxy resin composites by Zhu et al. [6, 7]. Allaoui et al. [8] studied the performance of MWCNTs in a rubbery epoxy matrix and concluded that there was considerable growth in Young's modulus and strength by adding up to 4 wt%. Upadhyay and Kumar [9] successfully improved the wear and friction characteristics of the CNT-added composites. In their studies, for nanoparticle-based epoxy composites, the test results for the friction coefficient range from 0.07 to 0.29 and the wear rate ranges from 10^{-3} to 10^{-2} mm^3/N-m for dry sliding condition. Also a significant enhancement in tribological features of the sample was observed with an increase in the concentration of filler up to

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5 wt%. Campo et al. [10] investigated the tribological performance of epoxy composites for different contents of CNTs and concluded that the best performance was shown by 0.5 wt% CNT composites. Three types of MWCNTs (NC3100 (long), NC3150 (short) and NC3152 (amino group-functionalized short MWCNT)) in weight percentages ranging from 0 and 0.5 were diffused to epoxy resin using calendering method. Compared to neat epoxy, the composite materials with MWCNTs exhibit a lower value for mass loss, coefficient of friction and wear rate. The influence of fabrication methods (tape casting and hot pressing) on tribological features of CNT-infused alumina composites (0 to 12 wt% of CNT content) was compared by Lim et al. [11]. For hot-pressed samples, the wear rate decreased with an increase in CNT up to 4 wt% and then increased with the addition of more CNT. The wear rate of the tape-casted composite materials was steadily decreasing with an increase in the addition of CNT up to 12 wt%. It was also noted that, with the addition up to 12 wt% CNT, there was a significant reduction in wear loss by maintaining a constant value of coefficient of friction. Moreover, the enhancement in tribological characteristics was due to the significant reduction in agglomeration for hot-pressed specimens.

Nowadays, application of CNT concentrates in the area of semiconductor and remote sensors besides automotive and aerospace [12]. Since the discovery of MWCNT [13] and single-walled carbon nanotubes (SWCNT) [14] by Lijima, the interest of fabricating CNT-based composite materials came into limelight. Polymers mixed with CNTs produce inimitable electromagnetic absorption [15] and electrical properties [16–20]. Considering the low production cost, better properties and easy availability, MWCNTs can be a best choice as reinforcement for the fabrication of composite materials [21–24]. Results from the existing literature indicate that a low percentage of MWCNT in epoxy matrix enhances the mechanical properties because of its greater aspect ratio [26, 27]. Furthermore, due to large surface area the MWCNT ensures better transfer of stress in the composite materials [28]. In order to get better mechanical properties, the combination of CNTs with different polymer matrixes like epoxy, polypropylene, polyurethane and polyamides had been seriously explored in the recent past [29].

In summary, there were several research carried out in connection with the effect of the MWCNTs on the performance of epoxy nanocomposites. Most of the reported work was based on mechanical performance of epoxy composites. The tribological performance in conjunction with mechanical properties analyses helps in selection of appropriate materials for most advanced engineering applications. In this work, the authors focussed on mechanical, tribological and morphological performance of MWCNT-based epoxy composites. The fabrication of epoxy nanocomposites was done using compression moulding technique, and the ultrasonication method was utilized for the dispersion of MWCNT in epoxy resin. The quantity of epoxy resin was fixed at 300 g, and the composition of MWCNT varied from 0 to 2 wt% of epoxy content. The fabricated composites with enhanced properties can be used in the field of aeronautics, electronic packaging, defensive shielding of computers, fuel cells and sporting goods.

Table 1 Properties of MWCNT nanoparticles

Properties	Range/Size
Diameter	40 ~ 50 nm
Length	2–10 microns
Surface area	250–270 m^2/g
Bulk density	0.06 ~ 0.09 g/cm^3

Table 2 List of epoxy composites prepared in the present study

Sample code	Material Combination
A	Epoxy resin (300 g) + MWCNT (0 wt%)
B	Epoxy resin (300 g) + MWCNT (0.5 wt%)
C	Epoxy resin (300 g) + MWCNT (1 wt%)
D	Epoxy resin (300 g) + MWCNT (1.5 wt%)
E	Epoxy resin (300 g) + MWCNT (2 wt%)

2 Materials and Experimental Methods

2.1 Materials

Resin and the hardener were blended to get the epoxy material. In this study, LY556 and HY951 were used as the resin and the hardener, respectively, with a proportion of 10:1. MWCNT (supplied by Platonic Nanotech Pvt. Ltd.) was used as the nanoparticle reinforcement. The properties of MWCNT nanoparticles are shown in Table 1.

2.2 Fabrication of Composites

300 g of epoxy resin was used for the fabrication of each composite. Steel moulds were used for preparing the composite material, which consists of a base plate and a top plate. The plates were cleaned using an abrasive paper of 1000 grit size and were further washed in acetone. Surfaces were then dried in a hot air oven for 10 min. The compositions of the nanocomposite materials formulated in this work are given in Table 2. In the initial step, the epoxy resin and MWCNT particles were mixed in definite proportion in an ultrasonicator for 2 h. The mixed resin–nanoparticle dispersion was then treated with a hardener in the proportion of 10:1. The blend of MWCNT and epoxy resin was poured continuously into a metal mould. The sample was further

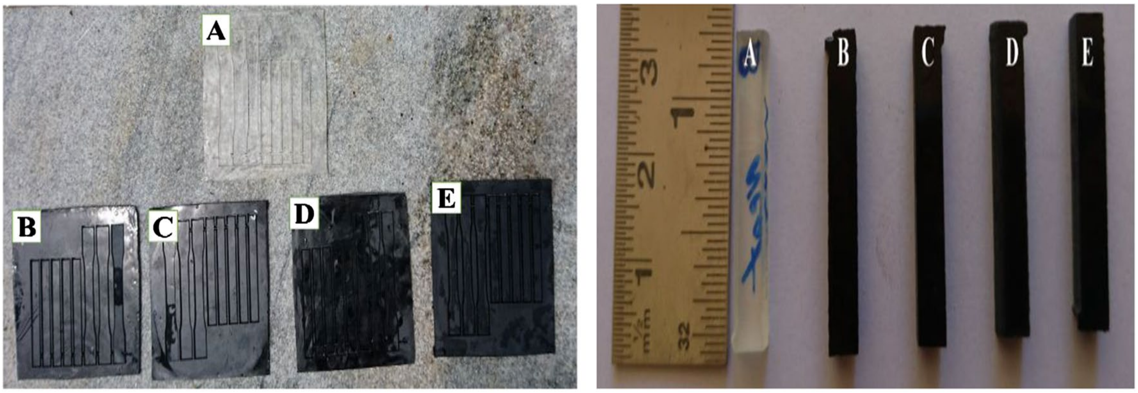
compressed for three hours using compression moulding machine. The dimension of the composite created was restricted to 200 × 200 × 5 mm. 5 samples of epoxy nanocomposites were fabricated as shown in Table 2 and Fig. 1a, as sample codes A, B, C, D and E. The specimens were cured at room temperature by applying uniform pressure on the mould using the compression moulding apparatus. After curing, for the purpose of various characterizations, these samples were sliced utilizing water jet machining in accordance with ASTM standards. The photographs of the prepared POD specimens are shown in Fig. 1b.

2.3 Mechanical Testing

The tests conducted as per ASTM standard for tensile and flexural properties, impact strength and hardness were detailed below. Under each test method, three specimens were tested. The average value was plotted and was used for the interpretation of the result and is explained in Figs. 3, 4, 5, 6 and 7. Error analyses was carried out for the experiments. The photographs of different mechanical test apparatus used are shown in Fig. 2a–d.

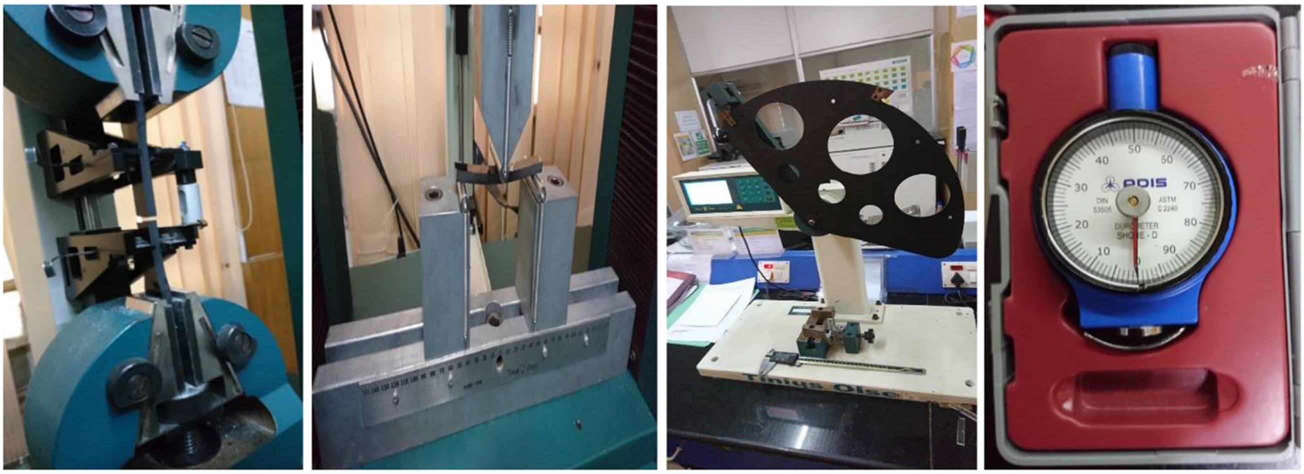
2.3.1 Tensile and Flexural Testing

The mechanical behaviour of the fabricated composites was tested using an universal tensile testing machine (Tinius Olsen H10KT) with a testing load range of 0 to10 ton. The experiments were conducted at normal room temperature. The tensile strength was determined as per ASTM D638. The flexural results obtained by three-point bending test were done with ASTM D790 standard.



A Prepared nanocomposites **B** POD specimens

Fig. 1 **(a)** Prepared nanocomposites, **(b)** POD specimens



A Tensile test **B** Flexural test **C** Impact Test **D** Hardness Test

Fig. 2 **(a)** Tensile test, **(b)** flexural test, **(c)** impact test, **(d)** hardness test

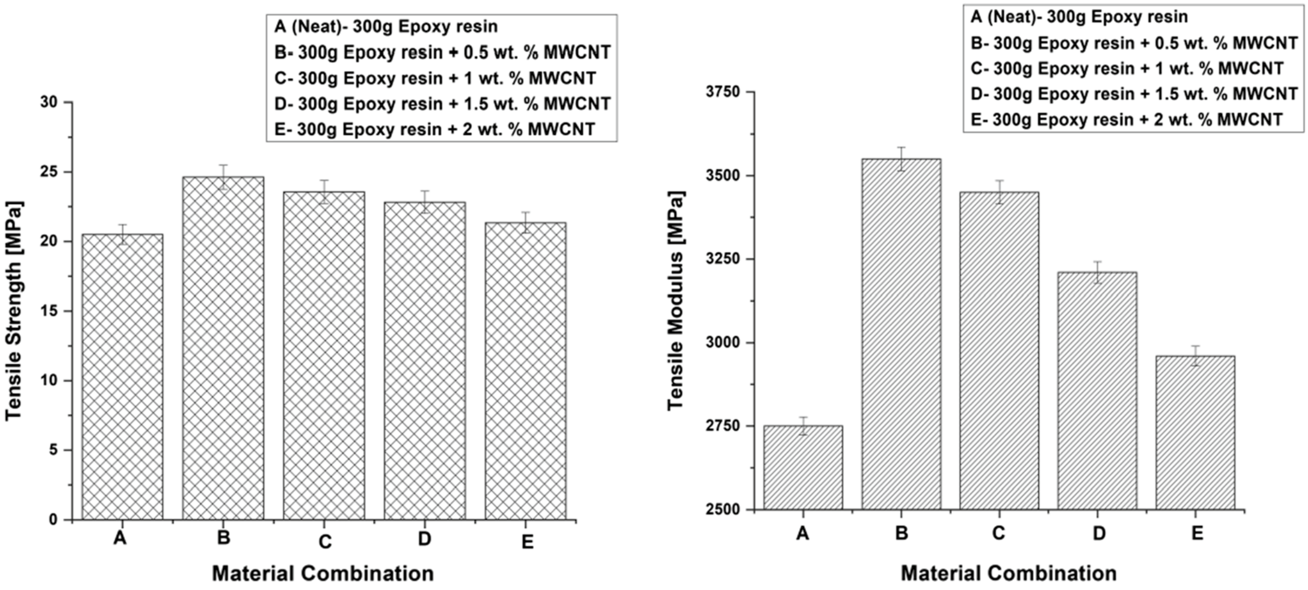


Fig. 3 Tensile strength and modulus of composites

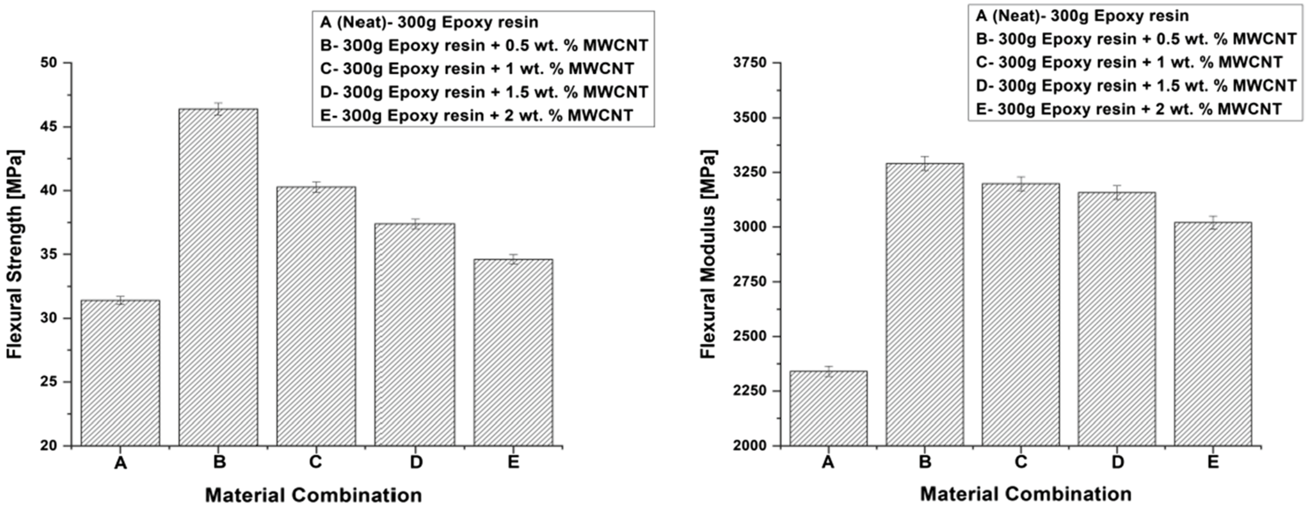


Fig. 4 Flexural strength and modulus of composites

2.3.2 Impact Testing

Impact strength for the composite specimens was done by an izod impact testing machine (Tinius Olsen IT 503) as per the ASTM D256 standard. To carry out the process, a cut with 2.5 mm notch depth was created on every specimen.

2.3.3 Hardness Testing

Shore D tester (Adis hardness tester) was used to carry out the test for hardness. Corresponding hardness value was found by penetrating the indenter foot of the durometer into the samples in accordance with ASTM D2240.

2.4 Tribological Testing

Tribological wear testing according to ASTM G99-05 standards was conducted by POD machine (Ducom-TR 20-LE). Composite specimens were used as the pin material, and the disc used here was made up of steel (EN31) with a hardness number of 60 HRC. For the purpose of experimentation, square pins of $5 \times 5 \times 30$ mm were utilized. The load applied ranges from 10 to 20 N at the surface interface.

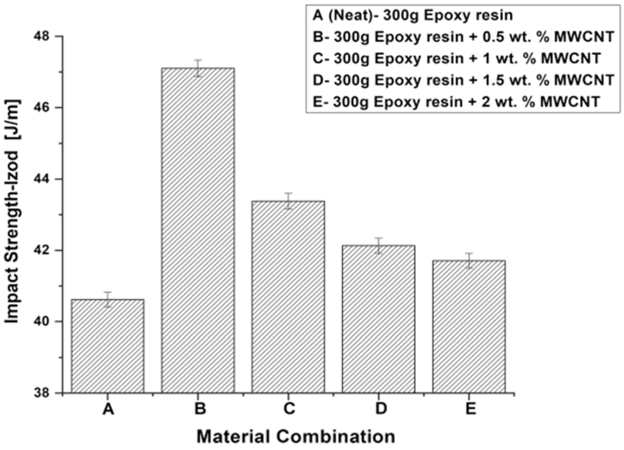


Fig. 5 Impact strength of composites

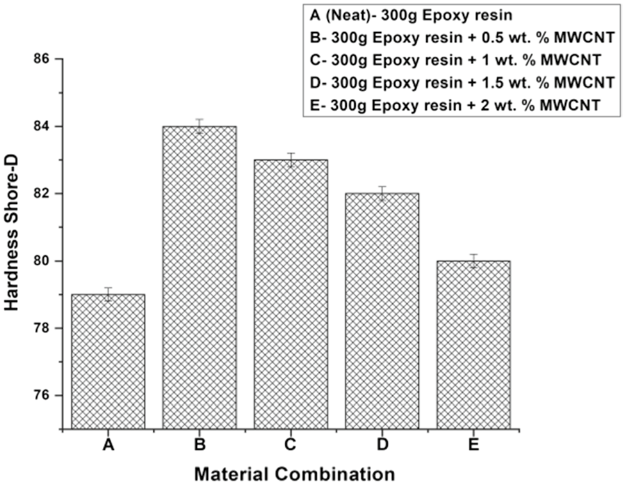


Fig. 6 Hardness of composites

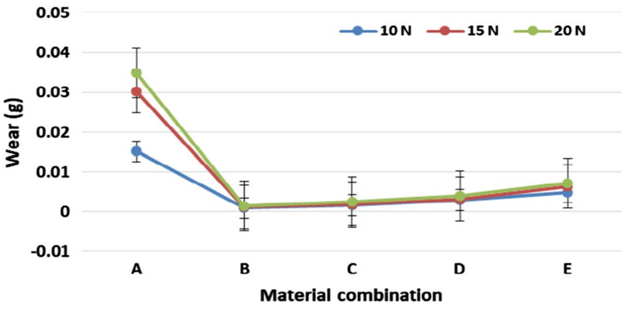


Fig. 7 Wear test at load 10 N, 15 N, 20 N

2.5 Morphological Analyses

The morphological analyses of MWCNT–epoxy composites were examined using SEM (Zeiss Sigma VP, USA) and TEM (JEOL JEM-2100, Japan). Morphology studies were conducted by scanning the samples at an accelerating voltage of 25 kV at a vacuum level of 1.5×10^{-3} Pa. SEM images were taken from the worn MWCNT–epoxy composite pin surfaces that were used in the wear test analyses. TEM analyses were carried out at 200 kV. Ultramicrotome (Leica EM UC7, Germany) was used to extract lean samples (90 nm thin slices) from the fabricated nanocomposites for TEM assessment.

3 Results and Discussion

3.1 Tensile Properties

The results of tensile strength and tensile modulus, respectively, of MWCNT–epoxy composites obtained are plotted in Fig. 3. The sample B shows higher tensile strength and modulus of 24.62 MPa and 3550 MPa, respectively. Comparing all other concentrations in the epoxy matrix, the greater tensile strength was achieved with 0.5 wt% of MWCNT. It was observed that a superior interfacial interaction was existing between the epoxy and MWCNT nanoparticles. Further 0.5 wt% MWCNT exhibits better nanotube dispersion with little agglomeration. The well-dispersed nanoparticles in the matrix can also be a reason for the observed enhancement in the tensile strength. However, concentrations beyond 0.5 wt% MWCNT in epoxy matrix exhibits non-monotonous behaviour due to agglomeration of nanotubes in epoxy composites and was supported by the TEM analyses discussed in Sect. 3.7 [21, 23, 24]. In summary, from the tensile results, maximum tensile strength and tensile modulus were 24.62 MPa and 3550 MPa, respectively, for 0.5 wt% MWCNT–epoxy composite (specimen B). The tensile strength and tensile modulus were increased up to 20.10% and 29.09%, respectively, by incorporating 0.5 wt% of MWCNT than the neat epoxy composite.

3.2 Flexural Properties

Figure 4 illustrates the test results of MWCNT–epoxy composite materials on their flexural strength and modulus. Sample B which was 0.5 wt% MWCNT provides higher flexural properties with 46.4 MPa and 3290 MPa as flexural strength and modulus, respectively. A betterment in flexural strength and flexural modulus had been noticed for nano-based epoxy composites with material composition B (0.5 wt% MWCNT) when compared with material composition A (Neat-0 g MWCNT). The stronger interfacial interaction between the resin and MWCNT may be the reason for the improvement in flexural strength for 0.5 wt% nanocomposite. The strong interfacial interaction between the epoxy and MWCNT nanotubes may aid to enhance the interlinear shear strength among the particles and was supported by the TEM analyses discussed in Sect. 3.7 [24–26]. In summary, from flexural results, maximum flexural strength and flexural modulus were 46.4 MPa and 3290 MPa, respectively, for 0.5 wt% MWCNT–epoxy composite (specimen B). The flexural strength and flexural modulus were increased up to 47.77% and 40.60%, respectively, by incorporating 0.5 wt% of MWCNT than the neat epoxy composite.

3.3 Impact Properties

The results obtained for the impact testing are shown in Fig. 5. A notch with 2.5 mm depth was incising on every specimen to carry out the process. The test reveals that sample B (0.5 wt% MWCNT) exhibits a higher impact strength of 47.10 J/m. It was remarkable to note that impact properties were in concurrence with tensile properties with the highest values for 0.5 wt% MWCNT–epoxy nanocomposites. The higher MWCNT increases the viscosity of the epoxy resin. Due to the larger specific surface area, the surface of MWCNT may not be fully covered with the epoxy matrix. This will affect the uniform allocation of nanotubes and the process of load transfer. It was supported by the TEM analyses discussed in Sect. 3.7 [16, 24, 26, 27]. In summary, from the impact results, the maximum impact strength was 47.1 J/m for 0.5 wt% MWCNT (specimen B). The impact strength was increased up to 15.95% by incorporating 0.5 wt% of MWCNT than the neat epoxy composite.

3.4 Hardness Properties

Figure 6 shows the result of hardness test. Corresponding hardness value was found by the penetrating indenter foot of the durometer into the sample. The higher hardness was shown by the 0.5 wt% MWCNT which was sample B, and the value obtained was 84 shore D. The higher crystallinity in nanocomposites and the inclusion of MWCNT in the epoxy resin were the causes of the superior hardness and were supported by the TEM analyses discussed in Sect. 3.7 [28]. The hardness of the nanocomposites can be increased by the homogeneous dispersion and interfacial collaboration in MWCNT [29]. From 0.5 to 2 wt% MWCNT composites, a decrement of hardness was also observed. This result particularly indicates that there was no proper dispersion due to the formation of clusters of CNTs at high-quantity levels of MWCNT, and as a result, the hardness will be reduced and also its efficiency as nanoreinforcement will also be reduced. In summary, from the hardness results, the maximum hardness was 84 shore-D for 0.5 wt% MWCNT (specimen B). The hardness was increased up to 6.33% by incorporating 0.5 wt% of MWCNT than the neat epoxy composite.

3.5 Wear Properties

POD wear test was conducted on a pin of size of 5 × 5 × 30 mm. Wear test for samples A, B, C, D and E was carried out for different loads (10 N, 15 N and 20 N). The observations are plotted in Fig. 7. According to Fig. 7, the maximum wear loss was observed for the neat epoxy (sample A, 0 wt% MWCNT) and the least wear loss was observed for the 0.5 wt% MWCNT epoxy resin composite (sample B). Weight loss for the sample B at load 10 N, 15 N and 20 N was 0.0009 g, 0.0011 g and 0.0014 g, respectively. Results show that the wear loss obtained for the neat epoxy was increased with the increase in load due to the thermal softening of the epoxy resin. The presence of cylindrical grapheme layers had self-lubrication property that will help in sliding or rotating motion. MWCNTs at the matrix surface will act as a tribo film which effectively lubricates the sliding surfaces. During the high load operation, the interfacial adhesion was poor which causes the MWCNTs to peel out very easily from the matrix and thereby reduces the lubrication effect of tribo film. Also, the dispersion of MWCNTs in epoxy resin efficiently transfers the stresses throughout the composites. Results show that 0.5 wt% MWCNT–epoxy composite provides superior tribological properties compared to all the other combinations for the load range investigated.

3.6 SEM Images of MWCNT–Epoxy Composites

Figure 8 depicts the SEM images of the surfaces of the pin after the test of tribological properties using POD. Secondary electron imaging (SE) was used to take the images to get a 200X magnification scale for a 1000 × 1000 µm image. Figure 8a portrays the surface of the pin after the sliding of the neat epoxy resin. It was observed a rough surface with a considerable quantity of burr development and wear detritus during the process of sliding which is shown in the image. Figure 8b–e shows the images of pin surfaces after sliding with 0.5 wt%, 1 wt%, 1.5 wt% and 2 wt% MWCNT–epoxy composites, respectively. From this, it was observed that the smoothest surface was obtained for the pin surface with 0.5 wt% MWCNT. It was also noticed that increase in the concentration of MWCNT from 0.5 to 2 wt%, the respective wear debris, deep shallow grooves, stepped fracture, plugging, crack and crack propagation were exhibited by the SEM images. As the concentration of MWCNT increases to higher levels, it was also observed that the adhesive wear dominates and results in an excessive weight rate. Figure 8c explains the crack propagation because of the aggregation of MWCNTs. Figure 8d exhibits a deep groove along with wear debris on the wear track. Figure 8e exhibits stepped fracture and plugging; this result could be due to their weak interaction with the MWCNT and matrix. Furthermore, it could be

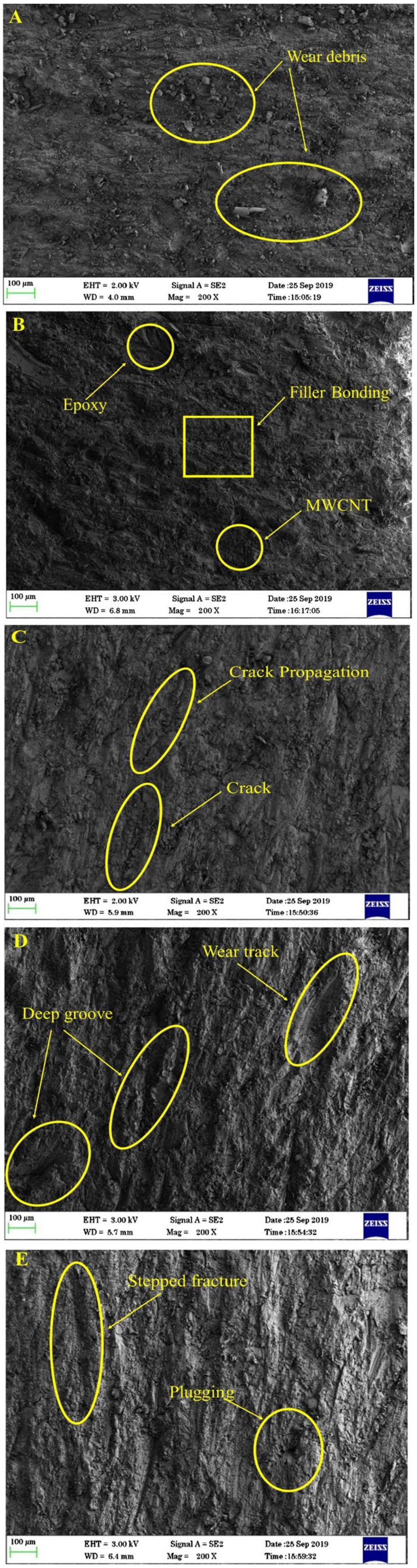


Fig. 8 SEM images of A, B, C, D, E nanocomposite samples after ▶ sliding of wear regions at 15 N

noticed that from the surface of the worn pin the quantity of materials peeled off was very little for nanocomposites compared with neat epoxy. It was also evident that the presence of plastic deformation and adhesion had been reduced for specimen B (Fig. 8b—0.5 wt% MWCNT), as observed from the SEM image. The formation of nanoclusters in the composite was not identified in the obtained SEM images of specimen B (Fig. 8b). So, it can be concluded that adding MWCNT was an effective method to diminish abrasion wear and to enrich the wear properties of epoxy–MWCNT composite. From the SEM analyses, it was clear that during the wear test, the applied load increases, and the damage to composites also increases. The analyses also show that undesirable damage to composites was detected at higher wear loads, whereas at lower wear loads only a little loss of matrix from the composite surface was observed.

3.7 TEM Images of MWCNT–Epoxy Composites

TEM had become extensively useful to examine the morphologies of MWCNT–epoxy composites. Figure 9b–e portrays the TEM images of composites. Figure 9b shows the uniform dispersion of carbon nanotube structures of MWCNT–epoxy composites, whereas Figs. C, D and E clearly depict agglomerated structures in the morphology of composites. Figures C, D and E show the dark region (as circled) which was the entangled cluster of CNT displayed in the TEM micrographs. With an increment in the amount of MWCNT, an increase in the number of clusters of entangled MWCNT was observed. It was observed that the viscosity of the composite increased with the addition of MWCNT compared with pristine epoxy resin. The increase in viscosity value was pointing to the poor dispersion of MWCNT in epoxy matrix. The intertwined MWCNT prevents the effective transfer of stress from matrix (epoxy) to reinforcement (MWCNT) during loading of composite which in turn leads to poor mechanical characteristics. As a result of intermolecular Van der Waals forces between the nanotubes, the formation of accumulated structures of MWCNT occurs. The easy entangling of MWCNT was due to the combinations of high aspect ratio and large surface areas. The MWCNTs were in intimate close contact with the polymer without any physical gap and are indicated by TEM on the MWCNT–epoxy interface of specimen B composite. After microtoming, no apparent MWCNT pull-out from the epoxy was noticed in the composite pieces and the greater part of the MWCNTs stayed in the epoxy, indicating the epoxy to MWCNT. Specimen B showed moderately uniform scattering with aggregations in the nm range while specimens C, D and E showed non-uniform scattering and arrangement of agglomerates of MWCNTs in the epoxy matrix. TEM images

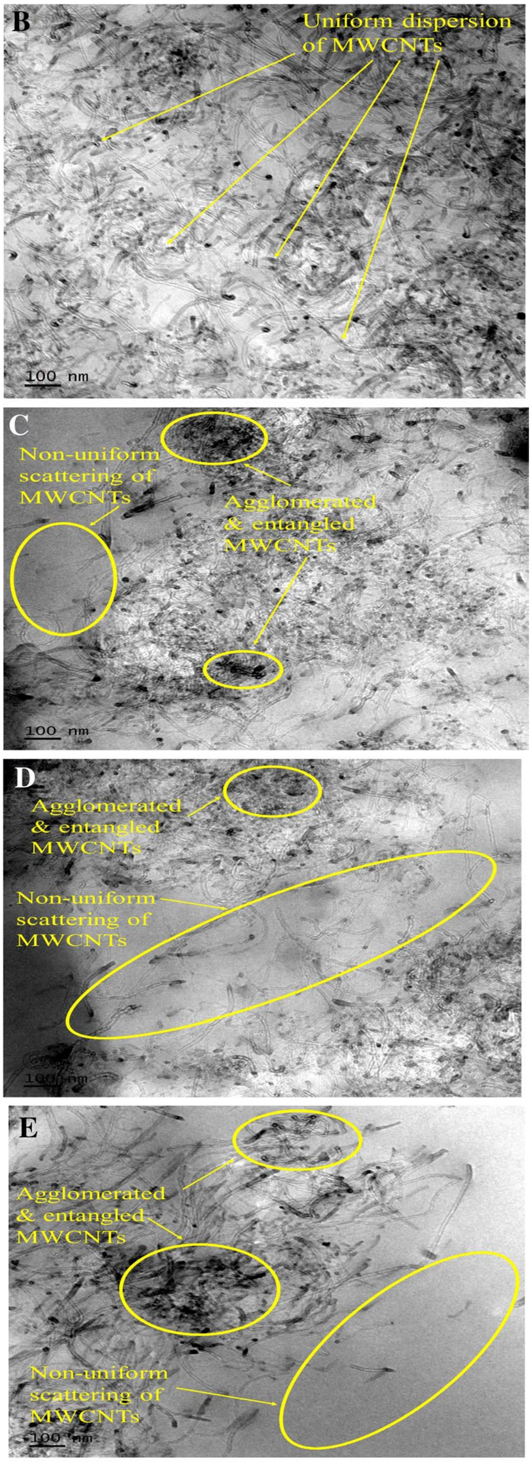


Fig. 9 TEM images of B, C, D, E nanocomposite

confirmed a decent bond between the MWCNT and the epoxy in the case of specimen B composite. In Specimen B composite, TEM results showed greater fondness between the composite's constituents and strong interfacial bonding impacts (buckled nanotubes). For composites of 1 wt%, 1.5 wt% and 2 wt% of MWCNT, several separated nanotubes can be detected.

4 Conclusions

Compression moulding technique was employed for the development of epoxy-based MWCNT composite materials. The significant observations obtained from various tests and characterizations were depicted below:

- The Specimen B (0.5 wt% MWCNT–epoxy nanocomposite) exhibited better mechanical, tribological and morphological performances.
- The comparison of tensile properties reveals that the combination 0.5 wt% MWCNT–epoxy composite has 24.62 MPa and 3550 MPa as tensile strength and modulus, respectively, which was higher.
- From the impact tests, it is observed that epoxy composite with 0.5 wt% MWCNT exhibits an impact strength of about 47.10 J/m and it is much greater than that of the neat composite which is only 40.62 J/m.
- Among the developed nanocomposites, 0.5 wt% MWCNT added epoxy resin exhibited the least wear loss. Wear loss was found to be increasing as we increase the percentage of MWCNT from 0.5 to 2 wt%.
- SEM images indicate that 0.5 wt% nanocomposites have the lowest abrasive wear, least number of wear debris and high filler bonding. The reason for the same is found to be the improved interaction developed between MWCNT and epoxy matrix.
- The superior affinity between interfacial bonding effects (buckled nanotubes) and constituents of nanocomposites is exhibited by 0.5 wt% MWCNT–epoxy composites. This observation is clearly evident from the morphological analyses using TEM.
- Based on the results obtained, the optimized nanocomposite can be recommended for a wide scope of use in the areas of aeronautics, electronic packaging, defensive shielding of computers, fuel cells and sporting goods.

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